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by carbonate of soda. The results were verified by determining the arsenious acid with permanganate of potash.

Arsenite of Zinc dried at 212°.

	Found.			Calculated.
	Sp. I.	Sp. II.	Mean.	
3ZnO* ..	55·29	54·74	55·01	55·23
AsO_3 ..	44·55	44·99	44·77	44·77
	99·84	99·73	99·78	100·00

The composition of the arsenite of zinc, like that of the arsenite of silver, establishes the capacity of arsenious acid for three atoms of a base of the formula MO.

Arsenite of Ammonia.

A salt of the formula $NH_4O.AsO_3$ having been described by Pasteur and by De Luynes, I was anxious to ascertain whether, in the presence of a large excess of ammonia, a more highly basic salt could be obtained.

Arsenious acid dissolves with difficulty in strong ammonia, but if water be added, the solution is effected much more rapidly, and on afterwards adding more strong ammonia, a precipitation of minute prismatic crystals takes place. These crystals were rapidly washed with alcohol, and dried by violent pressure between blotting paper, since they deliquesced and evolved ammonia very rapidly in the air. For analysis, the salt was weighed out in hydrochloric acid, the arsenic precipitated as sulphide, and the chloride of ammonium in the filtrate determined by evaporation. A verification for the ammonia was obtained by an alkalimetrical determination, and for the arsenious acid by permanganate of potash. The analysis fully corroborated the remarkable formula $NH_4O.AsO_3$ assigned by De Luynes.

	Found.		Calculated.
	De Luynes.	C. L. B.	
NH_4O	22·07	20·48	20·80
AsO_3	77·93	79·09	79·20
	100·00	99·57	100·00

* Zn = 32·7.

The existence of the following arsenites appears now to have been established.

Monobasic Arsenites.

$KO.AsO_3$ ..	Pasteur	C. L. B.
$NaO.AsO_3$ ..	„	„
$NH_4O.AsO_3$ ..	„	„ De Luynes
$BaO.AsO_3$ ..	Filhol	
$CaO.AsO_3$ ..	Simon	

Bibasic Arsenites.

$2KO.AsO_3$ ..	Pasteur	
$2NaO.AsO_3$ ..	„	
$2BaO.AsO_3$ ..	Filhol	
$2CaO.AsO_3$ ..	Simon	
$2MgO.AsO_3$ ..	C. L. B.	
$2PbO.AsO_3$ ..	Berzelius	C. L. B.

Tribasic Arsenites.

$BaO.2HO.AsO_3$ (?) ..	C. L. B.	
$2MgO.HO.AsO_3$ ..	„	
$2CaO.HO.AsO_3$ ..	Simon	
$2CuO.HO.AsO_3$ ..	C. L. B.	
$3PbO.3HO.2AsO_3$ ..	„	
$3ZnO.AsO_3$ ..	„	
$3AgO.AsO_3$ ..	„	Filhol

Sesquiarsenites.

$2KO.3AsO_3+3Aq$ ..	C. L. B.
$2NaO.3AsO_3$ ..	„

Acid Arsenites.

$KO.HO.2AsO_3+Aq.$ ..	Pasteur	C. L. B.
$KO.2AsO_3$		„
$NaO.2AsO_3+xAq.$ ..	„	„
$NaO.2AsO_3$		„

Although the composition of these salts leaves some doubt as to whether arsenious acid should be regarded as tribasic, I am much inclined to that belief by the following considerations—

The normal sulpharsenites of potassium and ammonium have, according to Berzelius, the formulæ $3KS.AsS_3$ and $3NH_4S.AsS_3$.

Phosphorous acid (PO_3) which should present a very close analogy to arsenious acid, has a tribasic character: for its crystallised hydrate has the formula $3HO.PO_3$; and although the phosphites of soda, baryta, and lead, are represented by Wurtz as $2NaO.PHO_4$, $BaO.HO.PHO_4$ and $2PbO.PHO_4$, they might plausibly be written on the tribasic type, as $2NaO.HO.PO_3$, $3aO.2HO.PO_3$ and $2PbO.HO.PO_3$.*

Boracic and arsenious acids are interchangeable in the class of tartrates known as *emetics*, so that if the former be really a triatomic group, as represented in a former communication, it is probable that the latter is so.†

I have not yet completed the examination of the arsenious ether obtained by decomposing one equivalent of terchloride of arsenic with three equivalents of sodium-alcohol; but I have every reason to believe, that it contains three equivalents of oxide of ethyl to one of arsenious acid.

XL.—*Description of a combined Maximum and Minimum Mercurial Thermometer.*

By W. Symons.

In 1860 I exhibited at the Pharmaceutical Society a double registering mercurial thermometer which had acted perfectly for some time, but soon afterwards that and other similar tubes were deranged by the mercury separating, thus destroying the usefulness of the instrument. When showing the first instrument to Dr. Redwood, he informed me that Dr. Macvicar had described a somewhat similar instrument at the Chemical Society; but, on reference, I found that while he used two fluids, his tube was straight, so that to have an open scale the thermometer must be inconveniently long: my instrument had an advantage in this respect, as by doubling the tube it was rendered more portable.

* Nitrous acid is commonly grouped with arsenious and phosphorous acids. Bromeis has described $3PbO.NO_3$ and $2PbO.NO_3 + HO$, whilst Pasteur has recently obtained $CuO.NH_4O.HO.NO_3$.

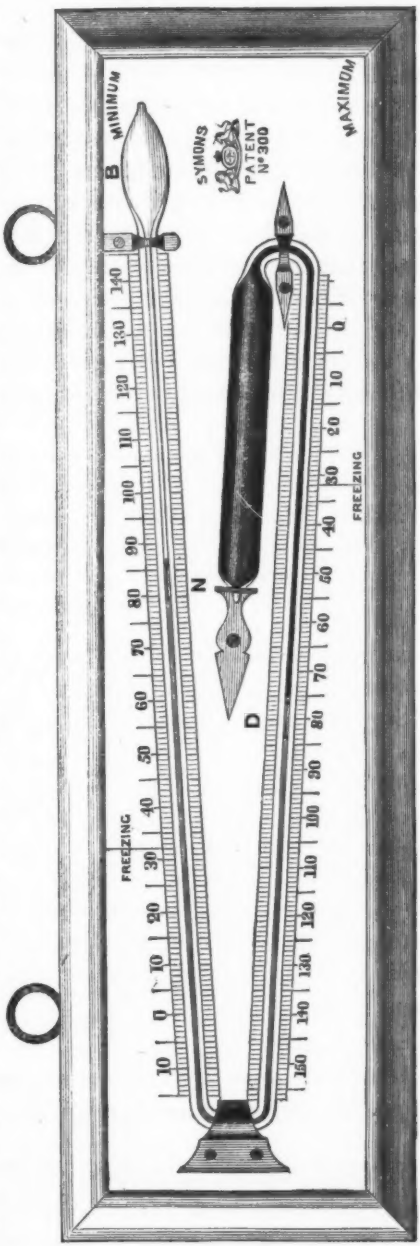
† It is worth notice that both boracic and arsenious acids form vitreous bi-acid salts with the alkalies.

From Dr. Macvicar's remarks (Journal of Chem. Soc., vol. xi, page 106) it is doubtful whether he had ever constructed a satisfactory instrument. He says,—"Between 20 and 30 years ago Mr. Adie, of Edinburgh, to prevent oxidation in Rutherford's maximum thermometer, introduced, above the mercury, naphtha; and I saw, the other day, one of these thermometers which, during that long interval, had preserved the mercurial column unbroken." Dr. Macvicar had no doubt tried some unsuccessful experiments in consequence, as he adds:—"It is certain, however, that it is not every kind of liquid that passes under the name of naphtha that will do so." My expeience would lead me to say, it is not any kind of such liquid that will do so. I have tried every such liquid that I could procure, and a variety of others kindly supplied to me by a Fellow of this Society, but without success.

After pursuing the subject for some time with alcohol, naphtha and various hydro-carbons, the thought suggested itself to me to experiment with a totally different class of liquids, selecting those with a high specific gravity. During the winter of 1860 and 1861 I had a variety of tubes exposed to all the frosts of that very severe season, and was happy to find some of these experiments much more successful. Sulphuric acid of various strengths, mixtures of sulphuric acid with sulphate of soda and alum, and a solution of common salt nearly saturated, stood the test without freezing and without the mercury being separated. Some of the original tubes are now shown, and also a thermometer with dilute sulphuric acid, made in January, 1861, it will be seen to have worked satisfactorily. Since that time, a great number have been made and sent successfully to all parts of the British Islands; but, bearing in mind the failure of my first attempt, I have waited until this arrangement has been fully and fairly tested before bringing it more distinctly before the scientific public.

As I have alluded to Dr. Macvicar's second instrument, I may be allowed perhaps to add that the first described by him in vol. x, p. 221, is very similar to a thermometer suggested long since by Dr. Traill, and described in the "Library of Useful Knowledge." In stating this, I would not for a moment insinuate that it was not an original idea of Dr. Macvicar's; for all who have the slightest knowledge of that gentleman will fully acquit him of appropriating to himself knowingly the suggestions of others; but it may be mentioned, as one amongst the many instances of the independent reproduction of similar inventions by different individuals.

The thermometer now shown consists of a tube bent as in the figure; the cylindrical bulb and tube up to D are filled with mercury, and from D to N with dilute acid; the upper bulb B and the tube to N are filled with air; at D and N are inserted steel indices covered with glass; the index D acts just as the index in Rutherford's maximum thermometer, and the index N as a minimum.



The advantages of this instrument, as compared with Rutherford's, are: 1st. That it combines the two instruments in one; 2nd. As the maximum index floats in a dense liquid, the thermometer is never deranged by the index sinking into the mercury; and, 3rd. It is virtually a mercurial minimum. The advantages over Six's form are: 1st. It is a mercurial thermometer; 2nd. The tubes being nearly horizontal, spring indices are not required; the tubes can, therefore, be smaller, and a frequent source of derangement, especially in carriage, is avoided; and, 3rd. The disadvantages and inaccuracies in extreme temperatures, either high or low, arising from the

high specific gravity of mercury, does not exist in the new instrument.

The indices are set either with or without a magnet, as most convenient.

My friend and namesake, Mr. G. J. Symons, of Camden Town, who is probably known to many connected with this Society, as a meteorologist, kindly undertook to compare the daily readings of one of these instruments with two others of the usual kind, supplied by different makers of repute; and the result will, I think, show that this instrument may be depended on even for scientific observations, the average discrepancies being under two-tenths of a degree, and these may be accounted for, to some extent, at least, by the respective bulbs being not exactly in similar circumstances with regard to passing currents of air.

XLI.—*Additional Notes on Reciprocal Decomposition among Salts in Solution.*

By J. H. Gladstone, Ph.D., F.R.S.

Since the publication of my paper on "Circumstances modifying the action of Chemical Affinity,"* and of "Some experiments illustrative of the reciprocal Decomposition of Salts,"† I have from time to time thought and experimented further upon the subject. The present communication is simply a gathering together of such observations as appear to me capable of throwing additional light on these laws of combination.

I reserve to some future occasion the experiments I have made on the time required to bring about a perfect state of equilibrium among various salts in solution, as the results are not yet sufficiently matured for publication, and the subject is a very distinct one.

The ultimate disposition of the various elements in solution is independent of the manner in which they were originally combined.

Suppose there be two basic elements M and M′, and two salt-radicles R and R′; the question is, whether if MR′ be added to

* Phil. Trans., 1855, 179. † Chem. Soc., Qu. J. ix, 144.

M′R in solution, the same distribution of elements will take place as if MR were added to M′R′. From the law of reciprocal decomposition it follows, as something like a corollary, that it must be so, and in my first paper I was content with one quantitative experiment; yet, as the opposite has been contended for, I thought it well to test the law in other instances.

The former experiment was this:—Two solutions were prepared, the one containing equivalent proportions of sulphocyanide of potassium, sulphate of potassium, and ferric nitrate; the other, equivalent proportions of sulphocyanide of potassium, nitrate of potassium, and ferric sulphate. These two mixtures, each containing the same absolute quantity of salts, were made up to the same volume with water; and the resulting colour was found to be identical, leading to the inference that the ultimate distribution of the four salts in each solution was the same.

The law was tested with a mixture of single equivalents of acetate of copper and nitrate of lead, and a mixture of single equivalents of acetate of lead and nitrate of copper. The colour was identical. A similar result was also obtained with sulphate of copper and nitrate of potassium, as compared with nitrate of copper and sulphate of potassium.

But it would rarely happen that a result perceptible to the eye could be obtained in this manner. The majority of metals that give colour to their compounds produce (unlike iron and copper) the same or nearly the same tint with whatever radicle they may be combined; and the same holds good with the colour-producing acids; whilst the great multitude of salts are colourless, and therefore invisible in solution. It occurred to me, however, that perceptible reactions might be obtained with these also, if mixtures were made of two colourless salts and then added to some coloured salt, such as ferric sulphocyanide, which is capable of reduction in colour by a redistribution of its components. The amount of alteration in this third salt would, of course, depend on the proportion of all the four salts ready to act upon it; indeed, the question would just be enlarged so as to include six elements instead of four —M, M′, and M″ with R, R′, and R″.

The experiment was tried in this manner:—Equivalent quantities of the sulphate and nitrate of potassium, and the sulphate and nitrate of magnesium, were dissolved in equal volumes of water; a mixture was made of equal quantities of the solutions of sulphate of potassium and nitrate of magnesium, and another mixture of equal

quantities of the solutions of sulphate of magnesium and nitrate of potassium: these were added to two equal portions of a solution of sulphocyanide of iron in the isoscope; and the diminution of the colour effected by them was observed. Now, the sulphate of potassium is known to have a much greater power of reducing the sulphocyanide than the nitrate has, but it was found that the two mixtures produced exactly the same tint.

In like manner, acetate of potassium with nitrate of lead produced the same diminution of colour (within the limits of probable errors of experiment) as acetate of lead and nitrate of potassium. The same two mixtures were tried also on ferric meconate, and gave a similar result.

The same experiment on the red sulphocyanide was also made with mixtures respectively of sulphate of copper with nitrate of of magnesium, and sulphate of magnesium with nitrate of copper. The same shade was produced by each.

A similar result was obtained with chloride of sodium mixed with sulphate of magnesium, and sulphate of sodium mixed with chloride of magnesium. These two mixtures were also tried on the deep scarlet bromide of gold, and they were found to produce an equal reduction of the colour. The two had the same effect likewise on a solution of the double iodide of platinum and potassium.

Two solutions were prepared, the one of the double sulphate of copper and potassium, the other of an equivalent of sulphate of potassium mixed with an equivalent of sulphate of copper. They produced the same effect on a solution of ferric sulphocyanide. This experiment is of importance, not because there was much doubt, *à priori*, that the condition of a double salt in solution is the same whether it has ever been crystallized or not, but because at first sight some experiments recorded by Professor Graham seem to point to the opposite conclusion. He found, in respect to the salt above mentioned, and the sulphate of magnesium and potassium, that the double salt was more diffusible than its mixed constituents.* Yet this seems to be the case only when the solutions are freshly prepared and in the cold, and the discrepancy probably arises solely from the slowness of the action by which uniformity is ultimately produced.

When there are several salts with the possibility of solid matter forming, the ultimate result may indeed be influenced by the order

* Phil. Trans., 1850, 22.

in which they are mixed. Thus, in the experiment above narrated, with chloride of sodium and sulphate of magnesium compared with chloride of magnesium and sulphate of sodium, it was only when the salts were mixed together beforehand that they equally reduced the iodide of platinum and potassium. When they were added one after the other to the red salt, a deposit of platinum separated in the one case, though not in the other. This, however, is not a real exception to the general rule, which only refers to salts actually in solution.

Extension of M. Margueritte's experiment.

M. Margueritte has shown,* by a number of instances, that if a salt MR is less soluble in water than another salt M′R′, or than MR′ or M′R, the addition of M′R′ causes a larger quantity of it to be dissolved. One instance is that of chlorate of potassium, which is more soluble in a solution of chloride of sodium than in pure water. Now, from the law of reciprocal decomposition (of which this is a result), it was foreseen that after a single equivalent of the more soluble salt M′R′ had exerted its influence, a second equivalent would produce an additional effect, though to a smaller extent, and so on. On trying the experiment, this was found to be the case. 129 grm. of chlorate of potassium and 59 grm. of chloride of sodium were taken as equivalent proportions.

Chlorate of Potassium.	Chloride of Sodium.	Water required for solution.	Decrease for each equiv. of Chlor. Sodium.
1 equiv.	0	2493 meas.	0
1 „	1 equiv.	2208 „	285 meas.
1 „	2 „	2060 „	148 „
1 „	4 „	1910 „	75 „

If a compound MR *is rendered more soluble by the presence of another compound* M′R′, *the addition of either* MR′ *or* M′R *will precipitate it from its saturated solution.*

This is also a necessary consequence of the law of reciprocal decomposition: for the MR′ or M′R will produce more MR, and the liquid is incapable of holding any more of this compound in solution. In a former paper, I examined one particular case

* Comptes rendus, xxxviii, 304.

of this general law, namely, when a salt insoluble in water dissolves in an aqueous solution of an acid; and the law was found to hold good in every instance except one. That apparent exception was in the case of ferric phosphate dissolved in hydrochloric acid, from which a precipitate was not obtained by the addition of phosphoric acid; but I have since found that phosphate of iron, freshly prepared by double decomposition, and well washed, dissolves in phosphoric acid, and thus the anomaly is fully explained. I have lately observed another apparent exception in the case of phosphate of calcium dissolved in hydrochloric acid, and this admits of another explanation, for the tribasic phosphate dissolves in three equivalents of the acid; therefore, if on the addition of phosphoric acid, any chloride of calcium be decomposed, the hydrochloric acid set free will suffice to dissolve the phosphate of calcium produced.

But in all these cases the compound MR was a salt scarcely soluble in water, and the more powerful solvent was an acid. Neither of these conditions is indispensable for the result. The following experiments will illustrate the general law in cases where 1st, the compound MR is soluble in water, though not so soluble as M′R′, M′R, or MR′; 2nd, where M′R′ is a neutral salt as well as MR.

1st—Sulphate of silver was dissolved in weak nitric acid; to a part of the solution nitrate of silver, and to another part sulphuric acid was added, with the production in each case of the crystalline sulphate.

Again, it was found that chloride of sodium had deposited from a solution of sulphate of sodium in hydrochloric acid; the liquid, which of course was saturated with the salt, and contained likewise free hydrochloric and sulphuric acids, and no doubt sulphate of sodium, was divided into two portions, and from each a crystallization of chloride of sodium was obtained by the addition, respectively, of sulphate of sodium and hydrochloric acid.

2nd—Where the solvent is a neutral salt. Chloride of lead was found to dissolve freely in acetate of sodium. Such a solution, saturated with the chloride, was prepared: it was divided into two parts; to the one neutral acetate of lead, and to the other chloride of sodium, was added, and in each case after awhile chloride of lead separated.

It appears, therefore, that the more general expression of the law deduced from theory, and given at the head of this section, is

confirmed by experiment. Yet it is not to be expected that in every case the precipitate will actually make its appearance; for the formation of a double salt or the special solvent powers of the compound added may be a disturbing influence, and may give rise to anomalous exceptions.

Action of Acid Solvents, and Reciprocal Decomposition in Alcohol instead of Water.

When ferric phosphate was dissolved in hydrochloric acid, the paleness of the solution, and the increase of colour on the addition of more acid, both rendered it evident to the eye that the whole of the iron was not in the state of chloride. Indeed, a comparison of the colour gave ground for the belief that in a saturated solution, at least as much as 85 per cent. of the iron is actually held dissolved as phosphate in the acid present.* It became interesting to see whether this solvent action would take place in the absence of water; and if so, whether the same proportion between the different salts would be maintained. For this purpose, a solution of hydrochloric acid in absolute alcohol was prepared, and its power of dissolving dry ferric phosphate was tried. It dissolved a great deal, assuming a pale yellowish-green colour. Thus, one question was answered; but there still remained the inquiry whether the proportions of the several salts in solution were the same as when the experiment was made in water. This received a reply in the negative, showing that the nature of the liquid influenced the reciprocal action, for a comparison of the solution with one of ferric chloride in absolute alcohol showed that a very large quantity—probably 95 per cent.—of the iron must have been present as phosphate. Again, the alcoholic solution became much darker when diluted with water, which is not the case with the ferric chloride itself dissolved in alcohol. Another experiment made with this solution of the phosphate was the following:—a portion was divided into two equal parts; to the one was added some of the hydrochloric acid dissolved in alcohol, to the other the same bulk of pure alcohol. At first the two appeared of the same colour, but after awhile that containing the large excess of hydrochloric acid became decidedly deeper.†

* See Chem. Soc., Qu. J., ix, 152.

† Some of the experiments described in this and the preceding section were brought forward at the meeting of the British Association at Leeds, in 1858, but the subject has been more fully worked out since.

Testimony from Diffusion Experiments.

I have already published* an account of an experiment showing that, from a mixture of equivalent proportions of chloride of sodium and nitrate of barium there diffused the four substances in such relative proportions as could only be explained on the assumption that each of the two acids had distributed itself between the two bases.† Professor Graham, in his recent paper "On Liquid Diffusion applied to Analysis," has described two experiments of a similar character; the one made with a mixture of chloride of potassium and sulphate of sodium, the other with equivalent proportions of chloride of sodium and sulphate of potassium; but the results are not so conclusive in favour of the law of reciprocal decomposition, for the numbers do not differ widely from such as might be given if all the chlorine were in combination with the potassium, and all the sulphuric acid with the sodium. Yet the accordance is not perfect, and the distribution of the four elements may be very different. One thing is conclusively shown by the perfect accordance of the two experiments, namely, that, in the words of Graham, "The acids and bases are indifferently combined, or that a mixture of chloride of potassium and sulphate of sodium is the same thing as a mixture of sulphate of potassium and chloride of sodium, when the mixtures are in a state of solution."‡

A Method of Quantitative Determination by means of Circular Polarization.

An argument in support of the law of reciprocal decomposition has already been drawn from an experiment made by Bouchardat,§ while examining the circular polarization of camphoric acid. He found that certain camphorates rotate the plane of polarization less than the acid itself, and that when one of these salts was supersaturated with hydrochloric acid, the solution did not exhibit so much polarizing power as it would have done had the whole camphoric acid been set free. Unfortunately, the experiment was not made with equivalent proportions of the different substances, and hence it has no quantitative value.

* British Association Report, 1860, and Chem. News, Aug. 11th, 1860.

† The relative proportions in the diffusate expressed in equivalents were—sodium 1,253, chlorine 1,175, barium 812, nitric acid 892.

‡ Phil. Trans., 1861, 197.

§ Comptes rendus, xxviii, 319.

It occurred to me, however, that interesting numerical results might easily be obtained by taking advantage of the fact that different compounds of the same body rotate the plane of polarization differently; and just to test the possibility of this, I have made some determinations with two substances belonging to very different groups—nicotine and tartaric acid.

Nicotine gives a strong left-hand rotation, but when combined with hydrochloric acid it entirely loses this power.* A solution of known strength gave a rotation of $-14°$: it was mixed with an equivalent proportion of chloride of ammonium; the two odours of nicotine and ammonia were perceptible in the mixture, and when it was examined with the polariscope it indicated only $-10{\cdot}5$.

Hence it may be concluded, that sufficient nicotine to produce the wanting $-3{\cdot}5°$ of rotation had entered into combination with hydrochloric acid displacing of course an equivalent amount of ammonia. These numbers happen to have the common divisor 3·5. The four substances must therefore have been present together in solution in very nearly, if not exactly, the following proportions:

$$\mathrm{Nic\,HCl + 3\,NH_4Cl + NH_3 + 3\,Nic.} \qquad (\mathrm{Nic = C_{10}H_7N.})$$

The experiment was repeated with chloride of sodium in place of chloride of ammonium. A rotation of $-28°$ was reduced to that of $-25°$ indicating the following composition for the mixture:

$$\mathrm{3\,NicHCl + 25\,NaCl + 3\,NaO + 25\,Nic.}$$

As the nicotine has decomposed less chloride of sodium than chloride of ammonium, and its absolute tendency to unite with the hydrochloric acid must have been the same in both experiments, it follows that the soda must have a greater tendency to combine with the hydrochloric acid than the ammonia has, as compared with their tendency to remain in combination with water alone. In combination with hydrochloric acid 3 equivalents of ammonia, in fact, balance themselves against the nicotine, and as many as 8·3 equivalents of soda are required to do the same.

It is evident that the above experiment might be repeated with innumerable other salts, and tables of respective affinity might thus be drawn up. The method is also available for experimenting on the influence of the quantity of any of the constituents.

Tartaric acid gave results which are not so easily understood. It has already been observed that equivalent proportions of the isomorphous tartrates of potassium and ammonium have an equal

* Indeed, when the acid was in great excess, Wilhelmy's observation of a slight indication of right-handed polarization seemed to be confirmed.

influence on the polarized ray, and the same appears to be true of the sodium-salt. The amount of rotation is increased by the alkali, and apparently independently of whether it exists in the state of the neutral or acid-salt: thus:—

Tartaric acid	.. ..	$=+10°$
„	+ 1 equivalent of sodium (bitart.)	$=+20{\cdot}5$
„	+ 2 equivalents „ (neut. tart.)	$=+31{\cdot}$

Tartaric acid was mixed with an amount of citrate of sodium, sufficient to form the bi-tartrate of sodium, if it should decompose it entirely. The polariscope showed that a partial decomposition had ensued. Additional portions of the citrate were added, and an additional production of the tartrate resulted in each case. The numbers were—

Tartaric acid	. .. .		$=+10°$
„	+ ½ citrate of sodium ..		$=+14{\cdot}4$
„	+ 1	„ ..	$=+18{\cdot}5$
„	+ 2	„ ..	$=+23{\cdot}$

This result is in strict accordance with what was theoretically expected: the sodium has distributed itself between the two acids in certain proportions; namely, 8·5 to the tartaric, and 12·5 to the citrate, when equivalent proportions were employed; and when more citrate of sodium was added, more tartrate was produced.

Now, citric and tartaric are analogous acids, and perhaps the above is the true explanation of the phenomena observed; but when other salts of sodium were employed, results were obtained which showed some effect on the polarized ray that is not to be accounted for by such simple decompositions. Thus, with acetate of sodium—

Tartaric acid	.. ..			$=+10°$
„	+ 1 equivalent of acetate of soda			$=+22°$
„	+ 2	„	„	$=+28°$
„	+ 3	„	„	$=+31°$

When two or three equivalents of the acetate were employed, the results obtained are intelligible enough, but why should a single equivalent of acetate of sodium produce a greater increase of rotation than a proportionate amount of hydrate of soda would have done?

But there are greater anomalies still. When nitrate sulphate,

or chloride of sodium, or chloride of ammonium is added to tartaric acid it actually reduces the rotating power. Thus:—

Tartaric acid	.. ..	$=+10°$
„	+ 1 equiv. nitrate of sodium ..	$=+\ 7°$
„	+ 2 equiv. „ „ ..	$=+\ 5°$
„	+ 2 equiv. sulphate of sodium ..	$=+\ 8°{\cdot}5$
„	+ 2 equiv. chloride of sodium ..	$=+\ 3°{\cdot}5$
„	+ 2 equiv. chloride of ammonium	$=+\ 4°$

With sulphate of ammonia, a very slight increase of rotation was obtained.*

The cause of these phenomena has not yet been ascertained. It evidently will interfere with the use of tartaric acid for the purpose intended; but, doubtless other substances besides nicotine might be found to give trustworthy indications.

XLII.—*On the general occurrence of Titanic Acid in Clays, and the method employed to estimate it; on the Analysis of Iron Ores, and Siliceous Minerals containing Iron, the separation of Oxide of Iron from Titanic Acid, and the methods of estimating Iron.*

By Edward Riley.

Some time since the author presented to the Society a short communication,† pointing out the presence of titanic acid in a series of Welsh fire-clays, and at the same time proposed a new test for the metal. On a subsequent occasion also he pointed out, in a verbal communication, the errors that may arise from not carefully testing for the above acid, which is present, as will subsequently be shown, in all clays,—or at any rate its absence is the exception.

It is my object to bring before the Society, not only the method adopted for determining titanic acid, but at the same time to

* Since the above was written, I have found that Biot has observed the remarkable changes in the rotation of the polarised ray by tartaric acid caused by dilution, or by combination with various other bodies. His experiments throw no light on the question of reciprocal decomposition.

† Quarterly Journal of the Chemical Society, vol. xii, page 13.

enter minutely into the various methods of analysis usually adopted with silicates, including also iron ores, and to notice some of the points which have, up to a recent period, been to me very unsatisfactory; it must, however, at the same time, be admitted that there are still many points which require investigation.

Most chemists who have made the analyses of clays and minerals of a similar description, will agree that when the analysis is conducted in the way ordinarily advocated, many of the processes are very unsatisfactory.

The general method adopted for the analysis of a clay is to take from 15 to 20 grns. and fuse it with an alkaline carbonate; the fused mass is then dissolved in hydrochloric acid; the solution evaporated to dryness; the mass heated; and the silica separated by re-solution in hydrochloric acid; the insoluble matter obtained is regarded as silica, without any further treatment, or by some chemists it is either treated with a solution of hydrofluoric acid, or exposed to the vapour of that acid in Brunner's apparatus. After the silica is entirely dissolved, it is evaporated to dryness and ignited. Each of the last two methods is open to the objection that the greater portion of any titanic acid that may be present with the silica is converted into a fluoride of titanium, which is volatile and consequently lost in the subsequent ignition. This may be considered to be one of the reasons why titanic acid has not been found by chemists in all clays, although, generally speaking, a small residue is left when the silica from an analysis is heated as above, and the titanic acid may be detected with the blowpipe. This residue also contains traces of alumina.

Proceeding with the analysis, and precipitating by ammonia and well washing the precipitate, it is dissolved by hydrochloric acid (on the filter), and treated with a solution of pure caustic potash. In this process it will be observed that when the quantity of iron is small, instead of obtaining the ordinary and characteristic precipitate of peroxide of iron, a precipitate is obtained of a much lighter colour, and evidently a very impure peroxide of iron, so impure that the author has always found it necessary to ignite the washed and dried precipitate of iron and alumina, and redissolve it in acid, which process serves to separate a certain amount of silica which is always left in solution after the evaporation to dryness, and may be separated thus, or by a second evaporation to dryness, as shown in subsequent experiments. After separation of the silica by either of the two methods men-

tioned, a much more satisfactory separation of the iron and alumina may be made by potash. The oxide of iron, after separation is dissolved in hydrochloric acid and reprecipitated by ammonia; it is then generally weighed and considered to be pure peroxide of iron; but it very rarely dissolves completely in hydrochloric acid, sometimes leaving a residue which retains the form of the oxide; sometimes, but more rarely separating gelatinous silica. This residue, on being crushed, forms a powder which defies filtration, and in most of these cases consists of titanic acid. This has been found to take place more particularly in the analyses of clays and blast-furnace cinders or slags.

The following are the analyses of the blast-furnace cinders from the Dowlais iron works, showing the average composition of the cinder for a week's working. The cinders were taken as follows: cinder was taken from each furnace every day, and portions of the cinders finely pulverised, 200 grains of each being weighed out daily and kept separate in a bottle, labelled with the number of the furnace. At the end of the week the pulverised cinder in each bottle was again intimately mixed and analysed. The Table gives the result,—the numbers at the top being the numbers of the furnaces in blast at the time; column, "average of 13 furnaces," gives the analyses of a mixture of equal weights of pulverised cinder taken from each furnace at different times on one day.

In all the above analyses, the iron could not be obtained very pure, the impurity most probably consisting of a small quantity of titanic acid. The iron given in the analyses of blast-furnace cinders is generally too high. In many cases no manganese is given, and unless great care is taken, it is very difficult to obtain the oxide of iron even in a tolerable state of purity. These analyses are thought to be of sufficient interest to insert in the paper, as they are most probably the most extensive series of analyses of blast-furnace cinders yet made.

Average Composition of BLAST-FURNACE CINDER (SLAG) *during a Week's working of the Furnaces.*

	1	3	4	5	9	10	11	12	13	14	15	16	18	*Mean.*	Average of 13 Furnaces.
Silica	39·09	42·52	41·30	44·88	42·08	43·68	42·45	41·08	45·23	42·57	40·02	40·69	38·48	41·85	43·07
Alumina	17·14	14·96	16·21	15·51	14·19	16·25	14·30	13·65	11·55	13·12	14·71	14·82	15·13	14·73	14·85
Protoxide of Iron	2·07	3·62	2·65	6·91	2·35	1·33	2·04	1·29	3·08	3·48	2·52	2·19	0·76	2·63	2·53
Protoxide of Manganese.	1·15	1·50	1·10	1·67	1·59	1·15	1·22	1·02	1·02	1·18	0·91	1·04	1·62	1·24	1·37
Lime	31·95	30·04	30·55	23·81	31·55	28·57	30·89	34·32	32·09	31·35	32·27	32·60	32·82	30·99	28·92
Magnesia.....	4·31	3·95	4·42	4·38	4·67	4·42	5·32	4·65	3·78	4·44	5·47	4·63	7·44	4·76	5·87
Potash.....	1·98	2·06	2·26	1·98	1·73	2·21	2·04	1·75	1·53	1·79	1·72	1·71	1·92	1·90	1·84
Calcium	1·64	0·85	1·20	0·59	1·37	1·29	1·04	1·22	1·04	0·89	1·31	1·29	1·23	1·15	1·01
Sulphur	1·31	0·68	0·96	0·47	1·10	1·04	0·83	0·98	0·83	0·71	1·05	1·03	0·99	0·92	0·89
Phosphoric Acid	·22	0·41	0·13	0·43		0·10	0·10			0·17	0·25		0·15	0·15	traces.
	100·86	100·59	100·78	100·63	100·63	100·04	100·23	99·96	100·15	99·70	100·23	100·00	100·54	100·32	100·35
Metallic Iron	1·61	2·81	2·06	5·37	1·81	1·03	1·58	1·03	2·39	2·70	1·96	1·70	0·57	2·04	1·97

Furnaces 1 to 16, Common White Forge-pig. 18 Furnace, on Foundry-pig.

Should the mineral also contain phosphoric acid, a portion of that acid is invariably left with the oxide of iron, although by far the larger portion is found in solution with the alumina. The alumina is separated by acidifying the solution and boiling with chlorate of potash, to destroy any organic matter then precipitated by carbonate of ammonia; the phosphoric acid may be separated, after the alumina has been weighed, by dissolving in hydrochloric acid, adding tartaric acid, ammonia, chloride of ammonium, and a magnesian salt.

It is rare that clays contain an appreciable amount of manganese; any that they may contain will be partly precipitated with the iron and alumina; this is one of the many objections to the precipitation by ammonia. The lime is then separated by oxalate of ammonia and weighed either as carbonate or sulphate; this determination appears to be quite satisfactory. The magnesia is next precipitated by ammonia and phosphate of soda. In clays this mode of precipitation is often very unsatisfactory, the precipitate being quite flocculent, instead of exhibiting the characteristic crystalline appearance of the ammonio-magnesian phosphate. This may be due in some cases to a little alumina held in solution by the large excess of ammonia used to precipitate the oxide of iron and alumina, but there is reason to think that it arises also from other causes which it must be admitted have not yet received sufficient attention to enable one to speak positively respecting them.

Having briefly sketched the ordinary method employed for analysing clays, &c., I shall proceed to describe some experiments made in relation more particularly to the estimation and separation of titanic acid.

First, in treating the silica with hydrofluoric acid, to prevent the fluoride of titanium from volatilizing, sulphuric acid should be used; by this means the whole of the titanic acid with the silica may be obtained. It it, however, invariably mixed with alumina and minute traces of iron, as will be seen by the experiments detailed.

More titanic acid may be separated by again evaporating to dryness the filtrate from the silica, heating it strongly, redissolving in acid, and separating insoluble matter which consists of a mixture of titanic acid and silica with traces of alumina.

The results of numerous experiments lead me to the conclusion that the higher the temperature to which the fused mass is heated,

after solution and evaporation to dryness, the greater will be the quantity of titanic acid left with the silica in the insoluble state, and therefore the smaller will be the quantity obtained in the second evaporation.

The oxide of iron obtained in the way just described in the analysis of clays, was tested as follows for titanic acid :—After it had been ignited and weighed, it was dissolved in hydrochloric acid, and insoluble matter was separated. This was burnt and treated in a platinum crucible with hydrofluoric and sulphuric acid, and the filtrate was reduced with a solution of sulphate of soda, added in large excess, sufficient to neutralise all the free hydrochloric acid. The solution was then boiled, and when nearly the whole of the sulphurous acid was expelled, a white precipitate was formed. The boiling was continued until a slight indication of sesquioxide of iron was seen ; the liquid was then rapidly filtered; the filter, which was not washed, was transferred wet into the platinum crucible containing the residue left on treating the mixture of silica and sesquioxide of iron with hydrofluoric acid,—then dried and burnt carefully at a low temperature ; and the burnt mass was fused with bisulphate of potash, dissolved in cold water, and boiled after perfect solution. Immediately on boiling, a precipitate was formed, which, on filtering and testing, gave all the distinct reactions of titanic acid. The following are the results obtained from the analysis of a Welsh fire-brick :—

Some considerable portion of the brick was pounded up ; and a small portion was reduced to very fine powder, 19·23 grns. of which after ignition, was taken for analysis. It was fused over a Bunsen's burner with 6 or 7 times its weight of pure carbonate of soda, dissolved in hydrochloric acid, evaporated to dryness, and carefully heated. The silica was separated by solution in hydrochloric acid and dilution with water. The silica obtained weighed 12·09 grns. Silica treated with hydrofluoric and sulphuric acids, evaporated to dryness and ignited, left residue ·225. This residue was fused with bisulphate of potash, and dissolved in cold water, and the liquid after complete solution was boiled. After boiling some time, a distinct precipitate was formed, but hardly sufficient to weigh. The solution precipitated by ammonia yielded a precipitate of alumina containing a trace of iron, which weighed 0·21 grn. The filtrate from the silica was evaporated to dryness and heated ; the residue dissolved as before in hydrochloric acid, and the insoluble residue separated: it weighed 0·36 grn. This

residue treated with hydrofluoric and sulphuric acids, evaporated to dryness and ignited, left residue = 0·16 grn. This residue was fused with bisulphate of potash, and dissolved in cold water, and the liquid after solution was boiled; immediately on boiling, a precipitate formed, which was boiled for some time, allowed to stand for a night in a warm place, then collected on a filter, washed with water containing sulphuric acid, and weighed 0·105 grn., and this gave with microcosmic salt the characteristic reaction of titanic acid. The filtrate from the titanic acid was precipitated by ammonia, and gave a precipitate of alumina, which weighed ·075 grn., considered as Al_2O_3. The filtrate from the second evaporation for alumina and sesquioxide of iron was made nearly neutral with ammonia, and a moderate quantity of acetate of soda was added; the solution well boiled, and the basic acetates of iron and alumina were collected and washed, a few drops of acetate of soda being added at each washing; the precipitate was dissolved in hydrochloric acid and precipitated by ammonia, then washed, dried, ignited, and weighed: its weight was 6·16 grns. This was dissolved in hydrochloric acid, a flat-headed glass stirrer being used to crush the lumps. After complete solution, a residue of silica weighing 0·04 grn. was left and separated; the solution of sesquioxide of iron and alumina in hydrochloric acid was treated with pure potash in a platinum dish; and after boiling, the sesqui-oxide of iron was separated by filtration.

The sesquioxide of iron, after re-solution and precipitation by ammonia, was ignited and weighed: its weight was ·675 grn. This was dissolved in hydrochloric acid, a residue retaining the shape of the pieces of oxide of iron and coloured with it then remaining; these lumps were crushed and the boiling was continued until the residue was perfectly white; it was, however, so small in quantity, that no weight could be obtained for it on burning the filter. The filtrate was treated with bisulphite of soda in sufficient quantity to neutralize the whole of the free hydrochloric acid; it was then boiled till sulphurous acid could only just be smelt. After application of heat, a precipitate soon formed which gradually increased. When this precipitate began to assume a yellow tint from oxide of iron, it was rapidly filtered, and the filter, without being washed, was thrown into a platinum crucible, dried and burnt, then fused with bisulphate of potash. After fusion, the mass was dissolved in cold water, and then boiled. After boiling some time, a precipitate was formed, which was allowed to stand a night, then filtered and

weighed as before described; the weight obtained was ·095 grn. The filtrate from the oxide of iron after treatment with potash, re-solution in hydrochloric acid, and re-precipitation by ammonia, was evaporated to a small bulk, and after treating with ammonia and phosphate of soda, gave indications of the presence of magnesia; $2MgO.PO_5$ obtained = 0·12 gr.

To separate alumina, the filtrate from oxide of iron was acidified with hydrochloric acid, chlorate of potash was added, the solution well boiled, and the alumina precipitated by ammonia and carbonate of ammonia, washed, dried, ignited, and weighed. Its weight was 5·535 grns. On dissolving it in hydrochloric acid, a similar quantity of silica was left = 0·03 grn. The filtrate treated with tartaric acid, magnesian salt, ammonia, and chloride of ammonium gave, after standing a night, a few distinct crystals of ammonio-magnesian phosphate, shewing the presence of a trace of phosphoric acid. The lime and magnesia were separated by the usual methods; with the magnesia there was a very small amount of the flocculent matter already alluded to. The lime weighed as sulphate gave 0·285 grn. The ammonio-magnesian phosphates weighed 0·56 grn.

The alkalies were determined by boiling the finely levigated brick with strong hydrofluoric acid in a gold crucible. 57·55 grns. taken after the brick had been thoroughly decomposed, was evaporated to dryness with sulphuric acid in a platinum dish; the mass, after being strongly heated, was well boiled in water, the lumps being crushed with a flat-headed glass rod; and the whole was transferred into a large beaker without filtration, and precipitated by ammonia and carbonate of ammonia. This method is adopted on account of the difficulty frequently experienced in separating the matter left insoluble in the boiling water, the fine particles usually clogging the filter. After separation of the precipitate by ammonia and carbonate of ammonia, the filtrate is evaporated to dryness, and ammoniacal salts volatilized; the residue is then treated with sulphuric acid and again evaporated to dryness, to drive off excess of sulphuric acid, then dissolved in water, treated with a solution of baryta in excess, and filtered. The usual method given is to treat with acetate of baryta, but it has always been found almost impossible to separate the sulphate of baryta by filtration, as it passes through the filter and filters very slowly; the use of hydrate of baryta entirely obviates this difficulty. The filtrate is acidified with acetic acid, evaporated to dryness and ignited; the carbonate of baryta and charcoal are separated by filtration; the carbonates

of the alkalies are dissolved in water; the solution is acidified with hydrochloric acid and the alkalies weighed as chlorides: the chlorides obtained weighed 2·09 grns. The above method has always been found to yield most satisfactory results, and is, moreover, expeditious.

The potash and soda were separated in the usual way with bichloride of platinum, and 5·73 grns. platinum salt obtained.

Silica.

Silica by first evaporation . .	12·09	
Al_2O_3 with silica separated by HFl.	0·21	11·88
Silica by second evaporation . .	0·36	
TiO_2 separated by HFl . . .	0·16	0·20
Silica with iron and alumina . .	0·04	0·04
		12·12

Brick		Silica				
19·23	:	12·12	: :	100	:	63·02

Silica 63·02%

Titanic Acid.

Titanic acid from silica, second evaporation .	0·105
Titanic acid from Fe_2O_3	0·095
	0·200

Brick		Titanic acid				Titanic acid
19·23	:	·200	: :	100	:	1·04

Titanic acid 1·04%

Alumina.

Alumina with silica		0·21
Alumina with titanic acid		0·075
Alumina separated from iron .	5·535	
*Less silica with alumina .	0·030	
	5·505	5·505
		5·790

* This silica is not calculated as coming from the brick: it is thought to be due to the action of potash on the glass used.

Brick		Alumina				Alumina
19·23	:	5·79	: :	100	: :	30·11

Alumina 30·11%

Sesquioxide of Iron.

Magnesia with oxide of iron weighed as $2MgO.PO_5$ 0·12

$2MgO.PO_5$		$2MgO$		$2MgO.PO_5$		MgO
113·34	:	41·34	: :	0·12	:	0·043

Titanic acid with iron	0·095
Magnesia „	0·043
	0·138

Fe_2O_3

$0·675 - 0·138 = 0·537$

Brick		Fe_2O_3				Fe_2O_3
19·23	:	0·537	: :	100	:	2·79

Lime.

$CaO.SO_3$		CaO		$CaO.SO_3$		
68	:	28	: :	285	: :	0·11735
If 19·23	:	0·11735	: :	100	:	0·61

Lime 0·61%

Magnesia.

$2MgO.PO_5$ from Fe_2O_3	0·120
$2MgO.PO_5$ from CaO.	0·565
	0·685

$2\ MgO.PO_5$		$2MgO$		$2MgO.PO_5$		MgO
113·34	:	41·34	: :	685	:	0·2498
Brick		MgO				MgO
If 19·23	:	0·2498	: :	100	:	1·29

Magnesia 1·29%

Alkalies.

$KCl.PlCl_2$		KO		$KCl.PlCl_2$		KO
244·2	:	47	: :	5·73	:	1·1028

Brick		KO				KO
57·55	:	1·1028	: :	100	:	1·92

KO		KCl		KO		KCl
47	:	74·5	: :	1·1028	:	1·748

Mixed chlorides KCl NaCl

2·09 — 1·748 = 0·342

NaCl		NaO		NaCl		NaO
58·5	:	31	: :	0·342	:	0·1812

Brick		NaO				NaO
57·55	:	0·1822	: :	100	:	0·31

Tabulated Results.

Silica	63·02
Titanic acid . . .	1·04
Alumina	30·11
Sesquioxide of iron . .	2·79
Lime	0·61
Magnesia	1·29
Soda	0·31
Potash	1·92
Phosphoric acid traces .	
	101·09

DETERMINATION OF SILICA AND TITANIC ACID IN FIRE BRICKS.

Bricks in fine powder ignited before Fusion.

	Brick taken Grns.	Silica per cent.	Titanic Acid per cent.	Description of Brick	First Evaporation.			Second Evaporation.			
					Silica	Residue from Silica by hydrofluoric and sulphuric acid.	Titanic Acid in Residue	Alumina in Residue	Insoluble Residue	Titanic Acid in Residue	
1	35·975	65·11	1·05	Stourbridge (Hickman)	23·815	0·81	0·255	0·59	0·545	0·125	
2	20·90	63·42	1·05	do. (Rufford)	13·33	0 355	0·120		0·330	0·100	Alumina
3	28·39	60·49	0·60	Newcastle (Lucas)	17·49	0·535	0·17	0·40	0·435		0·155
4	27·11	60·60	0·42	Newcastle (Stephenson)	16·56	0·470	0·115	0·315	0·51		0·170
5	22·145	55·86	0·67	do. (Ramsay)	12·49	0·365	*		0·32	*	
6	21·965	62·96	0·96	Wortley, Leeds (Ingham)	14·03	0·475	0·15	0·335	0·375	0·06	
7	23·31	62·39	0·69	Harwarden, North Wales	14·555	0·32	0·03	0·225	0·475	0·13	
‡ 8	26·92	64·52	0·50	Yellow London Clay	17·54	0·405	†		0·275	†	
9	28·89	91·84	trace	Ewell Brick, Surrey	26·98	0·655 (Alumina)			0·210 (Silica)		
10	21·70	94·33	trace	Dinas Brick, South Wales	20·87	0·635 (Alumina)			0·235 (Silica)		
11	23·43	75·16	trace	Black Alder, Devonshire	17·64	0·295 (Alumina)			0·305 (Silica)		

* In 5 the residues from evaporation 1 and 2 were weighed together, fused with bisulphate of potash, dissolved in cold water, and silica separated Silica = 0·245 ; titanic acid = 0·15.

† In 8 the same method adopted as in No. 5; silica obtained = 0·245, titanic acid = 0·135, alumina = 0·205.

‡ Clay dried at 300° Fahrenheit.

‡ Clay dried at 300° Fahrenheit.

TABLE showing the results of various analyses in which Titanic Acid was found but not determined, except in No. 2. The Titanic Acid was not, in this analysis, determined by fusion with bisulphate of potash and precipitation by boiling, and is a little too high. In analyses 4, 5, 6, and 7 no indication of Titanic Acid was obtained when treating the soils by the ordinary methods; but special examination of the Sesquioxide of Iron obtained in the analysis revealed its presence; and, moreover, it was found that the soils contained Iserine, which was readily separated with a magnet.

		1	2	3	4	5	6	7
1. American kaolin	Silica	59·21	27·75	55·16	48·53	49·17	47·10	48·01
2. Bastard ironstone, technically known as "Jack"	Titanic acid	*	*	2·19	*	*	*	*
3. Blue clay from Wiltshire	Alumina	27·77	17·02	21·88 FeO	18·77	18·85	18·23	19·13
4, 5, 6, and 7. Soils from the West India islands	Peroxide of iron	3·34	10·80	4·71				
	Magnetic oxide of iron ..	..	..	..	9·58	9·77	10·81	9·31
	Manganese (red oxide) ..	..	1·42	..	0·55	0·55	0·75	0·49
	Lime	0·41	26·11	2·62	8·03	8·30	8·26	7·59
	Magnesia	0·41	11·84	1·51	4·62	4·11	5·38	4·03
	Potash	1·91	0·39	2·22	0·27	0·29	0·20	0·30
	Soda	traces of with Lithia	..	..	1·82	2·08	1·82	2·06
	Phosphoric acid	..	3·85 Sulphuric acid	.. Carbonic acid	trace			
	Sulphate of lime	..	0·51 Sulphur	2·40	0·20	0·12	0·20	0·20
	Combined water	6·87	0·14	6·04	3·33	3·12	3·24	3·85
	Moisture	0·24	0·15	..	3·24	2·35	2·49	4·20
	Organic matter	..	..	0·67	1·16	1·68	1·63	1·90
		100·16	99·98	99·40	100·10	100·39	100·11	101·07

* Titanic acid was detected in very appreciable quantity, but not determined.

Table I shows the amount of titanic acid found in ten samples of fire-bricks and in a sample of ordinary London clay. The titanic acid was determined in the way already detailed; viz., evaporating the filtrate from silica to dryness, and heating. The method employed is perhaps not very accurate, as most probably in all cases the whole of the titanic acid is not separated, even by two evaporations to dryness, as will be seen by referring to the detailed analyses: some was found with the iron. It, however, shows the general occurrence of this acid in clays; in many the amount certainly exceeds one per cent. It will be observed that in columns 3 and 4 the whole of the titanic acid is with the silica; in some cases it has been obtained entirely in the second evaporation. This appears to be due to the temperature to which the fused mass is heated after solution and evaporation to dryness. The higher the temperature, the greater is the quantity of titanic acid obtained with the silica, and *vice versâ*.

Professor Miller, of King's College, London, has found 1·25 per cent. of titanic acid in Stourbridge clay, and Mr. D. Campbell has found it in several clays, by adopting the method above detailed.

The last three bricks are essentially silica bricks, and contain no clay. Practical men have informed me that the last brick is of inferior quality.

In these experiments, my evaporating dishes were coated on the interior with the peculiar metallic lustrous stain which so pertinaciously sticks to all vessels in which a titanate is dissolved in an acid or precipitated by boiling.

Preparation of pure Titanic Acid.

Pure titanic acid was prepared from the red nitride crystals from an old blast-furnace hearth; it was obtained unintentionally. The crystals, after having been freed as far as possible from iron by means, first, of dilute sulphuric acid, then of strong hydrochloric acids, were heated with a mixture of nitric and hydrochloric acids: the consequence was, that some considerable portion of the crystals was oxidized. The titanic acid was separated from the crystals by decantation, then washed and fused with bisulphate of potash, and thrown down from the solution of the fused mass by boiling. The washing of this precipitate was interminable, and the sulphuric acid could not be completely washed out. On drying it and igniting, some sulphuric acid was driven off, and on treating

the ignited residue with water, no sulphuric acid was detected. From the above it would appear there is a difficultly soluble compound of sulphuric and titanic acid, which is continually washed out from the acid; and this is most probably the reason why the determination of titanic acid by precipitation from a boiling solution is not accurate.

Special Experiments with Titanic Acid.

2·78 grns. pure titanic acid were treated with hydrofluoric acid, and some sulphuric acid was added; the whole was evaporated to dryness, ignited, and weighed. Weight obtained 2·76 grns. It was then fused with bisulphate of potash, the fused mass dissolved in cold water, the solution boiled, and the titanic acid separated with the usual precautions. The titanic acid thus obtained weighed 2·535 grns. The filtrate, precipitated with ammonia, yielded a further quantity of titanic acid, weighing 0·12 grn., thus showing that the precipitation by boiling is not quite complete, but nearly so.

Experiments with Titanic Acid and Alumina.

Some pure alumina was prepared from alum by precipitating with ammonia, washing the precipitate, drying and igniting, re-dissolving in hydrochloric acid, and separating insoluble matter (a little silica), then re-precipitating by ammonia, washing, drying, and igniting.

Alumina ·22 and titanic acid ·08 were fused together with bisulphate of potash, the fused mass was dissolved in cold water, and the solution boiled. The titanic acid did not precipitate immediately, but in five or ten minutes; the alumina did not appear to affect the precipitation of the titanic acid.

No. 2. Experiments were made with 0·2 and 0·3 grn. of titanic acid with some alumina: the acid could always be detected, and alumina had no influence whatever on its precipitation. In fusing alumina with bisulphate of potash, care should be taken not to heat the mass too strongly, and drive off too much sulphuric acid, as in that case the fused mass becomes difficultly soluble in water, and in some cases, after solution, the alumina may possibly be precipitated by boiling from insufficiency of sul-

phuric acid. The precipitate cannot, however, be mistaken for titanic acid, and may be readily taken up by adding a little dilute sulphuric acid.

No. 3. *Quantitative experiment.* 1·135 alumina and 0·40 titanic acid were fused together with bisulphate of potash; the mass was dissolved in cold water, the solution boiled for some hours, and the titanic acid separated as usual, TiO_2 obtained = 0·325. The alumina was precipitated from the filtrate with ammonia and carbonate of ammonia, the liquid filtered, the precipitate washed once or twice, re-dissolved in HCl, and re-precipitated. The quantity of alumina obtained was 1·185—

TiO_2	0·325
Al_2O_3	1·185
	1·510

thus showing a loss on the titanic acid and a gain on the alumina, as might be anticipated from previous experiments.

Experiments with Titanic Acid and Sesquioxide of Iron.

Pure sesquioxide of iron was obtained from protosulphate which had been recrystallised three times from distilled water, by oxidation with nitric acid and precipitation by ammonia. Great difficulty was, however, experienced in dissolving the ignited sesquioxide of iron in the bisulphate of potash by fusion; some of the pure protosulphate of iron was therefore used after it had been pounded and thoroughly dried between folds of bibulous paper.

Exp. 1. 6·30 grns. protosulphate of iron and 0·28 grn. titanic acid were fused with bisulphate of potash, dissolved in cold water and boiled; no precipitate was formed at first, but after ten or fifteen minutes' boiling, a precipitate began to form, which gradually increased in quantity, and was coloured with oxide of iron.

Exp. 2. 6·59 grns. protosulphate were fused with 0·15 titanic acid and bisulphate of potash as before. The mass was dissolved in cold water, and the solution boiled. After boiling several hours no precipitate was produced. After the liquid had boiled down to a small bulk in a covered beaker, it was filled up with water and boiled again for a considerable time, but no precipitate was produced.

Exp. 3. 4·29 grns. protosulphate of iron and 0·12 of titanic

acid were fused with bisulphate of potash, dissolved in cold water, and boiled. After boiling for several hours, there were indications of the formation of a precipitate, but certainly nothing satisfactory, and not sufficient to indicate titanic acid, unless that substance was previously known to be present there. From the above experiments it is obvious why titanic acid has often been overlooked by chemists, and that oxide of iron should always be separated before testing for it.

Experiments with Titanic Acid and Ammonia.

2·425 grns. titanic acid were fused with bisulphate of potash, dissolved in cold water, and precipitated by ammonia; the precitate was washed, dried, ignited, and weighed, gave 2·58.

2·745 grns. of titanic acid were fused with bisulphate of potash and 200 grns. of pure chloride of ammonium added to the solution; it was then precipitated by ammonia, washed, dried, and ignited: it weighed 2·775.

From the above experiments, it is obvious that titanic acid is completely precipitated by ammonia, and that ammoniacal salts do not at all influence its precipitation.

My subsequent experiments with titanic acid relate more to its presence in iron ores and pig iron.

The method usually given in text-books for analysing iron ores or minerals containing much iron, is to dissolve them either in hydrochloric or nitro-hydrochloric acid, and separate the siliceous matter,—or, if the silica is in the soluble state, to evaporate the solution to dryness, re-dissolve in acid, and, after separating the silica, to precipitate the filtrate by ammonia. The precipitation by ammonia is open to many objections, which, in most analyses, are fatal to the accuracy of the results. These objections are, the precipitation of some manganese with the iron; the precipitation of the phosphoric acid as phosphate of lime, and of some carbonate of lime and magnesia. The precipitation of these last two bases may, in a great measure, be prevented by using great care; but not so with the former. An extensive experience in the analysis of iron ores and similar minerals has proved to me that phosphoric acid and manganese are very rarely indeed absent, phosphoric acid existing from a mere trace up to 4 per cent.

The difficulties above described may be obviated by nearly neutra-

lising with ammonia the filtrate from silica or the insoluble residue (the latter may be either analysed separately, or the silica separated by fusion, and the second filtrate added to the first), then adding acetate of soda or ammonia in excess, and boiling the solution for some time: the oxide of iron and alumina are thereby completely precipitated, together with all the phosphoric acid, which is entirely free from manganese, lime, and magnesia. This process also completely precipitates titanic acid if present in the ore. The precipitate must be washed with boiling water, and a few drops of acetate of soda or ammonia added every time it is washed: if this is not done, the precipitate clogs the filter, the washing becomes tedious, and some of the iron passes through the filter. This precipitate should then be dissolved in hydrochloric acid, re-precipitated by ammonia, dried, ignited, and weighed. Experience has proved to me that this is necessary, as in all cases the precipitate contains silica, which is, in a great measure, separated by re-dissolving the ignited precipitate in acid. The filtrate from the small amount of silica may be treated with potash, and alumina separated by acidifying with hydrochloric acid the filtrate from the oxide of iron, boiling with chlorate of potash, and adding carbonate of ammonia: the precipitate is then to be ignited and weighed. This alumina contains by far the larger proportion of phosphoric acid, but not all, as some is invariably left with the iron. The phosphoric acid may be separated from the alumina by dissolving it in hydrochloric acid and adding tartaric acid, ammonia, sal-ammoniac, and a magnesian salt. The oxide of iron is re-dissolved in hydrochloric acid, precipitated by ammonia, and weighed: if tested for phosphoric acid, it will always be found to contain that acid, if present in the ore. Should titanic acid be present, the oxide of iron is, after dissolving in hydrochloric acid, reduced with bisulphate of soda, the excess of sulphurous acid is boiled off, the solution nearly neutralised with ammonia, acetate of soda or ammonia added in excess, and the solution boiled. The titanic acid is then completely precipitated, and may be obtained pure by fusing with bisulphate of potash as before described.

The filtrate from the precipitate by acetate of soda is treated in a flask with bromine and ammonia, and heated gently, then boiled, whereby the whole of the manganese is most completely and readily separated as peroxide, which may be filtered off rapidly and weighed, after washing and drying, as Mn_3O_4. The filtrate is heated in the ordinary manner for lime and magnesia. When

an iron ore is analysed as above described, the sesquioxide of iron is not obtained in a pure state. The separation of the sesquioxide from alumina by potash is most unsatisfactory; and it will be found that the oxide of iron is too high, no reliance can be placed on weighing iron as sesquioxide after it has been separated from alumina by potash. In proof of this it will be necessary to detail some special experiments made on the determination of iron, the results of which prove that the only sure and safe method of determining it is by a standard solution either of bichromate of potash or permanganate of potash; or rather, it is never safe to consider sesquioxide of iron as such, until it has been dissolved and treated either by Penny's method or Marguerite's.

Careful experiments were made with pure sulphate of iron, the same being used as previously described. 6 pints of solution of bichromate of potash was prepared by dissolving 270 grns. of the salt in distilled water, and 140 grns. pure carbonate of potash added to make it alkaline; 1500 grns. burette was used. A standard solution was prepared by dissolving the finest bright-drawn iron wire in dilute hydrochloric acid in a 30 oz. flask closed with a funnel; after perfect solution of the iron wire, it was filled up with cooled boiled distilled water, making in all rather more than a pint of solution.

1 Iron wire 6·525 grs. required meas. solution 1285 ∴ 1000 = 5·0778
2 „ 7·075 „ „ 1393 ∴ 1000 = 5·0789

With the above solution, the iron was determined first in the pure protosulphate; then in the sesquioxide obtained from the protosulphate, after it had been precipitated by ammonia, and weighed as Fe_2O_3.

Iron by Standard Solution of Bichromate of Potash.

No. 1.	$FeO.SO_3$	30·66	required measures	1220	= iron per cent.	20·207
„ 2	„	34·615	„	1375	„	20·172
„ 3	„	48·265	„	1925	„	20·189
„ 4	„	27·29	„	1087	„	20·227
					Mean	20·199

Theory gives 20·144 per cent.

The protosulphate of iron was dissolved in water; the solution acidified with a moderate quantity of hydrochloric acid, and

diluted with rather more than a pint of cooled boiled distilled water.

Iron weighed as Fe_2O_3.

No.	1.	$FeO.SO_3$	33·155	gave	Fe_2O_3	9·575 = iron	per cent.	20·215
„	2	„	50·04	„	„	14·45	„	20·213
„	3	„	40·28	„	„	11·655	„	20·254
„	4	„	30·22	„	„	8·735	„	20·233
							Mean result	20·229

The sulphate of iron was dissolved in water, peroxidized with nitric and hydrochloric acid, precipitated by ammonia, dried, ignited, and weighed.

Iron determined in Sesquioxide by Standard Solution of Bichromate of Potash.

The above sesquioxide was dissolved in strong hydrocholoric acid, transferred into a 30 oz. flask filled two-thirds full with distilled water, and about as much sulphite of soda added as would cover a shilling, the liquid being gradually heated, and boiled till no smell of sulphurous acid was discernible, then cooled in water and treated with the standard solution.

No.	1.	Fe_2O_3	9·575	required	meas. solution	1315 = iron	per cent.	20·141
„	2	„	14·45	„	„	1991	„	20·141
„	3	„	11·655	„	„	1604	„	20·12
„	4	„	8·735	„	„	1200	„	20·165
							Mean	20·142

In dissolving the sesquioxide of iron in acid, some gelatinous silica separated in every case; it was, however, very minute in quantity, but distinctly discernible.

Comparing the results obtained by determining the iron directly by standard solution, determining the iron by weighing as Fe_2O_3, and again determining iron in the Fe_2O_3 by reduction and standard solution, we have—

	By standard solution direct.	Weighing as Fe_2O_3.	Determining. Fe in Fe_2O_3.
Iron per cent.	20·199	20·229	20·142

Theory 20·144.

From the above experiments, it is obvious that with a pure salt of iron, containing no other base, the determination of iron by

weighing as sesquioxide is accurate, and that its determination by standard solution of bichromate of potash, either in the pure proto-salt, or in the peroxide of iron from it, agrees in every respect with the former.

When, however, potash is used, or any quantity of fixed base is in solution, the determination of iron by weighing as sesquioxide is inaccurate; this will be seen by the following direct experiments. Moreover, the results of a very long experience have proved to me that the sesquioxide of iron obtained in an analysis of an iron ore is always too high; and it is very rare indeed that the determination of iron by weighing as Fe_2O_3, can be made to agree with the determination by standard solution.

Experiments with Sesquioxide of Iron, and its determination when mixed with Alumina and Titanic Acid.

Pure sesquioxide of iron, the same as prepared from the protosulphate previously alluded to, was used, and a standard solution of bichromate of potash, 1000 grns. of which were equivalent to 5·28 of iron.

10·39 grns. pure sesquioxide, dissolved in hydrochloric acid, and precipitated by ammonia, gave 10·435 Fe_2O_3. This re-dissolved in hydrochloric and reduced with sulphite of soda, required 1386 measures of the standard solution of bichromate, equal to 10·454 F_2O_3.

Separation of Sesquioxide of Iron and Alumina by Potash.

The potash used for the following experiments was carefully tested, and on operating on some quantity, traces of silica could be detected; it was, however, an inappreciable quantity. A little alumina was also detected, but too small in quantity to affect the results; the same oxide of iron and alumina, prepared as described, were used. Sesquioxide of iron 14·47 and alumina 2·225 were weighed out and dissolved in strong hydrochloric acid: the solution was transferred to a large platinum bowl, and well boiled; the sesquioxide of iron separated by filtration, dissolved in hydrochloric acid, re-precipitated by ammonia, then dried and ignited, weighed 14·705 grns. This was re-dissolved in acid, and found to contain a quantity of silica too minute to be weighed; the solution was reduced with sulphite of soda; the iron determined by standard solution required 2142 measures.

1000 measures = 4·75 = Iron 10·174 or Fe_2O_3 14·53.

The filtrate from the oxide of iron, after separation by potash, was acidified with hydrochloric acid, and well boiled with chlorate of potash, then precipitated by carbonate of ammonia, and the precipitate was well washed, dried, ignited: it weighed 2·195, which, on re-dissolving in acid, left no residue of silica.

Taken.	Obtained by precipitation.	Obtained by standard solution.	Excess.
Fe_2O_3 14·47	14·705	14·53	0·275
Al_2O_3 2·225	2·195		

Assuming the above experiment to represent the analysis of an iron ore, and that 20 grns. had been taken, the difference in the actual amount of sesquioxide of iron present and that obtained would be $0{\cdot}275 \times 5 = 1{\cdot}375$, or a difference of more than one per cent. in the amount of iron.

Separation of Sesquioxide of Iron and Titanic Acid.

Fe_2O_3 taken 14·51 ; TiO_2 0·21. The titanic acid was fused with bisulphate of potash, dissolved in cold water, and added to the dissolved solution of the oxide of iron in strong hydrochloric acid; the mixture was precipitated with ammonia, dried after washing, and ignited: it weighed 14·765. On dissolving it in hydrochloric acid, no indications whatever were observed that titanic acid was present, as the oxide of iron dissolved completely. The sesquichloride of iron was diluted with water, and reduced with sulphite of soda, the excess of sulphurous acid being boiled off; the solution was nearly neutralised with ammonia, and acetate of soda added in excess; the liquid was then boiled; the titanic acid was precipitated, together with a little sesquioxide of iron, and was rapidly filtered off; the precipitate of the filter was not washed, but dissolved in hydrochloric acid; the iron again reduced as before, and the titanic acid precipitated by nearly neutralising with ammonia and adding excess of acetate of soda. The titanic acid was thus obtained nearly free from iron. After filtering, without washing the filter, it was transferred into a platinum crucible, dried, then burnt, fused with bisulphate of potash, dissolved in cold water, and the titanic acid was precipitated by boiling, and collected on a filter, with the precautions already described. The quantity obtained was 0·20 grn. The sesquioxide of iron was determined by adding all the filtrates together, oxidizing with nitric acid, and precipitating by ammonia. The quantity

obtained was 15 grns.; this, on being re-dissolved in acid, was accidentally lost.

In this case, the excess was: 15·00 — 14·51 = 0·49. Every care was used to have the sesquioxide of iron perfectly washed.

Separation of Sesquioxide of Iron, Alumina, and Titanic Acid.

Taken Fe_2O_3 11·08; Al_2O_3 2·58; TiO_2 0·20; the oxide of iron and alumina were dissolved in hydrochloric acid; the titanic acid was fused with bisulphate of potash, dissolved in water, and added to the solution of iron and alumina. The whole were then precipitated together, and weighed 14·07 grns. On dissolving this acid no indication whatever of titanic acid was obtained, but there was left a slight gelatinous residue of silica, which weighed ·08. This was separated and treated with hydrofluoric acid and a little sulphuric acid, but left no residue; the filtered solution was boiled with pure potash in a platinum dish, and sesquioxide of iron separated. The alumina determined in a similar manner to that described in experiment 1, weighed 2·485 grns.; when re-dissolved in acid it left no appreciable residue of silica. The sesquioxide of iron on the filter, together with the titanic acid, was re-dissolved in acid and re-precipitated by ammonia, washed, dried, and ignited; it weighed 11·63 grns.; this, on re-dissolving in acid, left a residue of 0·15 silica, which gave no appreciable residue on treating with hydrofluoric and sulphuric acid. The solution from the silica was reduced with sulphite of soda, and treated in precisely the same manner as in experiment 2. The titanic acid weighed 0·20 grns. The filtrates from the separation of titanic acid by the acetate were mixed together, peroxidized with nitric acid, and the sesquioxide of iron was precipitated by ammonia; it weighed 11·215 grns., and was found to contain a trace of silica. The last filtrate from titanic acid was not added to the first two filtrates, as it was found, on testing it, to contain only a trace of iron, which did not appear quite pure sesquioxide. The sesquioxide of iron, after solution in acid, was treated with a standard solution of bichromate of potash, of which it required 1645 measures. 1000 = 4·75 = Fe 7·813, or Fe_2O_3 11·16. In this case, the excess in the oxide of iron is not very great; it is, however, occasionally obtained pure, or rather, nearly so; but this is the exception.

Having detailed the above experiments, which prove the errors that arise from weighing iron as sesquioxide, and considering it

pure without testing it by standard solution, I shall now give the results of an analysis of an iron ore, very carefully made, the results being in duplicate; and at the same time to show the very grave errors which arise from precipitating the iron and alumina in an iron ore by ammonia. A dispute arose some time since concerning the yield of an iron ore, in which samples were sent to an eminent provincial chemist and myself; but no agreement could be made on the yield of the samples; it was therefore agreed to take a large sample, have it dried, finely pulverized, and send a portion to each. The samples were identically the same in a fine powder. The analyses made bore not the slightest resemblance the one to the other, as seen by the following Table, the differences being entirely due to the dangerous practice of precipitating the solution of the ore by ammonia.

The method of analysis already described was adopted, and the following results were obtained :—

	grns.
Hygrometric water.	
No. 1. 38·54 grns. of ore lost, after drying at 212°, Fahr. .	1·21
„ 2. 38·09 „ „ .	1·24
Water, total amount, combined and hygrometric, determined by heating, in a glass tube drawn out at end, and absorbing water in a chloride of calcium tube :—	
No. 1. 21·145 grns. of ore yielded of water at a red heat	2·14
„ 2. 18·170	1·80
No. 1. By the action of hydrochloric acid (with little nitric) on the ore :—	
24·52 grns. of ore, dry at 212°, Fahr., gave of insoluble residue	4·55
„ Soluble silica	0·06
„ Sesquioxide of iron, alumina, and phosphoric acid, weighed together	15·25
„ Contained soluble silica	0·02
„ Sesquioxide of iron	13·84*
„ Sesquioxide of iron contained phosphoric acid .	0·175
„ Alumina	1·55
„ Alumina contained phosphoric acid. . .	0·47
„ Manganoso manganic oxide	0·05
„ Sulphate of lime	3·10
„ Pyrophosphate of magnesia	0·89

* Silica separated from sesquioxide of iron ·06 not calculated in analysis.

ANALYSIS OF OOLITIC IRON ORES.

	1.	2.
Clay and sand	18·80	18·42
Organic matter	0·60	0·60
Iron pyrytes	0·35	0·45
Peroxide of iron	49·97	47·98
Protoxide of iron	5·36	5·31
Oxide of manganese	0·20	0·44
Alumina	5·09	6·48
Lime	5·20	5·02
Magnesia	1·32	1·32
Phosphoric acid	1·71	1·88
Carbonic acid	4·61	4·93
Combined water	6·92	7·15
	100·13	99·98
Metallic iron, per cent.	39·39	37·95
Clay and sand contained—		
Silica	16·47	15·77
Alumina	1·28	1·27
Peroxide of iron	0·34	0·24
Potash	9·28	0·28

Precipitated by Ammonia.	3".	Results compared. 3.	4.
Extraneous water	19·20	13·70	7·03
Water of composition	11·07	41·99	37·71
Metallic iron	33·93	17·94	15·58
Oxygen	14·50	18·99	18·61
Silica	15·35	3·29	9·12
Carbonate of lime	2·66	2·79	6·48
Alumina	2·26	0·54	1·32
Magnesia	0·44	trace.	1·80
Phosphoric acid	slight	—	0·32
Oxide of manganese	—	—	0·76
Excess of carbonic acid	—	—	0·60
Organic matter	—	—	0·40
Iron pyrytes	—	0·73	—
Loss	0·59	99·97	99·73
	100·00		

1, 2, 3 and 4 dry at 212° Fahrenheit.

	grns.
The insoluble residue gave of—	
24·52 grns. silica, containing a trace of titanic acid .	3·91
„ Sesquioxide of iron	·085
„ Alumina	·315
„ Traces of lime and magnesia	
Analysis 2. By the action of hydrochloric acid, with a little nitric, on the ore :—	
20·74 grns. of ore, dried at 212° Fahr., gave insoluble residue	3·76
„ Sesquioxide of iron, alumina and phosphoric acid, weighed together	12·745
„ Soluble silica in above	0·02
„ Iron determined in the above by standard solution of bichromate of potash : measures required, 1600 = Fe_2O_3	11·017
„ Standard 1000 measures = 4·82Fe.	
„ Determination of phosphoric acid. Mean results % 1·80; therefore 20·74 grns. of ore contain PO_5	0·37
„ The total quantity of oxide of iron and alumina, &c. 12·745 grns. diminished by Fe_2O_3 11·017, PO_5 0·37 and SiO_3 0·02, leaves alumina .	1·338
„ Manganoso-manganic oxide	0·090
„ Sulphate of lime	2·490
„ Pyrophosphate of magnesia	0·74
The insoluble residue gave of—	
Silica	3·10
Iron and alumina 0·41, containing Fe_2O_3 .	0·05
Alumina by loss 0·36—0·10 silica contained in this precipitate	0·26

Determination of Iron direct.

19·75 grns. of ore, dried at 212°, required 1556 measures of standard solution of bichromate of potash, whereof 1000 = 4·82 Iron = ...	37·97 p. c.
23·90 grns. of ore, dried at 212°, required 1877 measures of standard solution of bichromate of potash, whereof 1000 = 4·82	37·85 „
From above analysis	37·78 „

Determination of FeO by dissolving the ore in a large flask in a current of pure carbonic acid, diluting with cold boiled distilled